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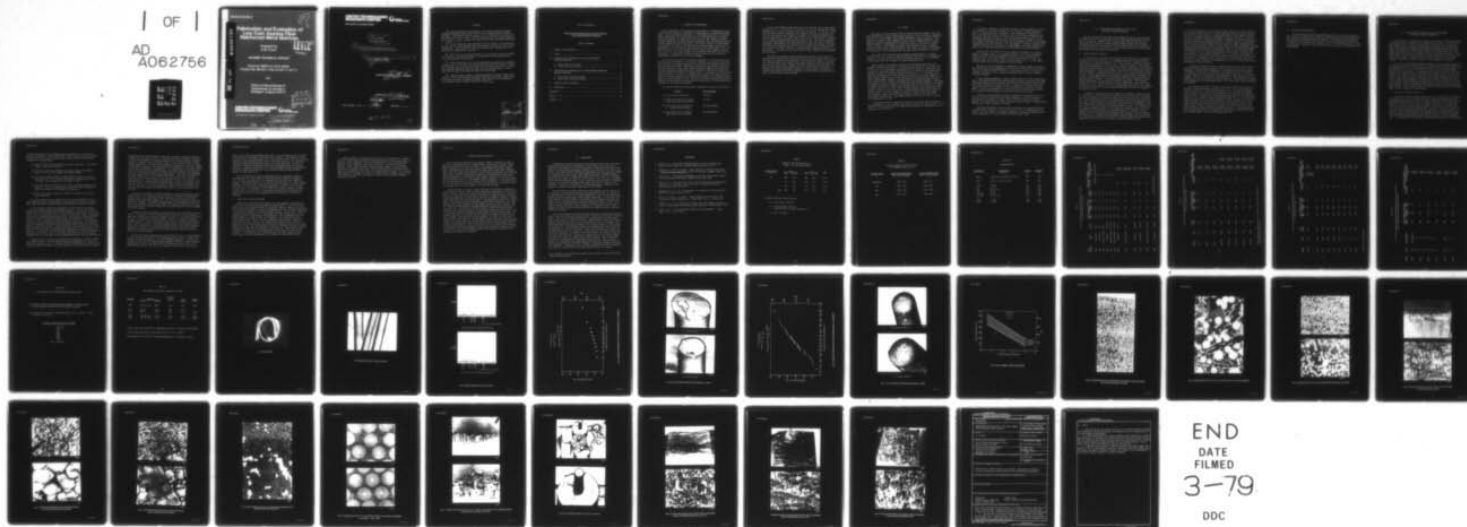
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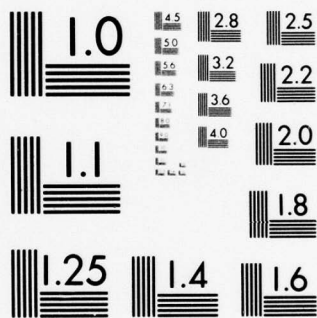
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# Fabrication and Evaluation of Low Cost Alumina Fiber Reinforced Metal Matrices

Prepared by  
K.M. Prewo

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## INTERIM TECHNICAL REPORT

Contract N00014-76-C-0035  
Project No. NR 031-784/9-23-77 (471)

for

Office of Naval Research  
Department of the Navy  
Arlington, Virginia 22217

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Fabrication and Evaluation of Low Cost  
Alumina Fiber Reinforced Metal Matrices.

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# ABSTRACT

This report describes the second phase of a program conducted at United Technologies Research Center under Office of Naval Research Contract N00014-76-C-0035. The technical contract monitor is Dr. B. MacDonald and the principal investigator is Dr. K. M. Prewo. The work described covers the period March 1, 1977 to August 1, 1978. During this period the following have been accomplished.

1. Two new fibers have been introduced into the program. Both fibers are SiC with one in a large diameter (140 $\mu$ ) monofilament form and the other a multifilament yarn. This second fiber has only recently been developed and is produced from an organometallic precursor

2. The new SiC yarn was characterized as to its surface chemistry, mechanical properties, fracture morphology, and compared with the characteristics of the large diameter monofilament.

3. Powder metallurgical and casting techniques were explored for composite fabrication and it was found that the PM approach offered the greatest success. Low cost, high rate processing techniques were also successfully used to produce high strength composites using both high and low bonding pressures. In the later case the matrix was densified while partially molten.

4. Composite axial strength, transverse strength, and impact energy were measured. The SiC monofilament reinforced composites offered the highest levels of strength and impact resistance; however, the strength of the fiber-matrix bond severely limited composite transverse strength.

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Fabrication and Evaluation of Low Cost Alumina  
Fiber Reinforced Metal Matrices

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## I. SUMMARY AND INTRODUCTION

This second phase of the program has been devoted to broadening the range of metal matrix composites available for consideration in engineering applications. As in the previous phase (Ref. 1), the emphasis has been on new fiber-matrix combinations that lend themselves to low cost fabrication procedures. This was undertaken in recognition of the fact that many composite systems, although possessing excellent mechanical properties, are not economical because of their significant fabrication cost. This effort involved the introduction of an entirely new fiber to the metal matrix composite scene, as well as the development of a new process of fabrication for a well-known older fiber. The former fiber is a SiC yarn obtained from the Nippon Carbon Company of Japan, while the latter is a large diameter SiC monofilament available from the Avco Corporation. Although both of these fibers are basically SiC, they differ considerably in properties and structural makeup.

As a first step, both fiber types were examined as to their tensile properties and surface chemistry. The surface condition, as was shown in the prior year's effort, is extremely important in controlling fiber-matrix wetting during composite fabrication. Both fibers were found to have highly carbon rich surfaces, which were later shown to make casting in aluminum alloys difficult. A surface oxidation procedure was then instituted at UTRC which altered this surface chemistry and removed the excess carbon. In addition, a surface treated version of the SiC yarn was also requested and obtained from Nippon Carbon Co. This treated fiber appeared to also have been subjected to an oxidation process similar to that practiced at UTRC. Another fiber surface modification process, which entails plating the fiber surface with a metal prior to infiltration with aluminum, is also currently underway.

The composite fabrication procedures included four types as listed below.

<u>Process</u>	<u>Fibers Studied</u>
(1) Liquid Metal Infiltration	SiC Yarn
(2) Powder Metallurgy with Liquid Phase Sintering Under Pressure	SiC Yarn
(3) Tape Fabrication Followed by Low Pressure Quick Bonding	SiC Monofilament
(4) Tape Fabrication Followed by High Pressure Quick Bonding	SiC Monofilament

In all cases aluminum alloys were used as the matrices and included compositions of Al + Li, Al + Mg, Al + Si and Al + Cu. All four processes presented opportunities for success, however, not without difficulty. It was only in the third and fourth process categories that composite mechanical properties thus far have been achieved which might be attractive for engineering application. Data are presented in this report which illustrate that high levels of strength can be achieved using processing times of only 15 min. This permits rapid use of hot pressing equipment and would significantly lower the cost of composite components. Unfortunately the inclusion of low pressure processing, by raising the consolidation temperature above the alloy solidus, did not maintain these high levels of strength. However, in selected large component applications the availability of low pressure may prove desirable and a compromise in performance may be possible.

The other two processes used were primarily directed at achieving a SiC yarn reinforced material and were limited in scope by the fragile nature of the yarn and the extremely small dimensions of the interfiber spacing. At present the casting approach still seems to offer great potential for future use; however, the SiC fiber surface chemistry has not yet been altered sufficiently to achieve fully uniform fiber distribution after liquid metal infiltration. In contrast, the powder metallurgical approach of process number two permits a uniform fiber-matrix distribution, however, low consolidation pressures must be used to avoid excessive fiber breakage.



## II. FIBERS

Two forms of silicon carbide fiber were used during the program. They differed extensively in their characteristics and also in their method of manufacture. The first is a 140 $\mu$  diameter monofilament supplied by Avco Corp. which is fabricated by the chemical vapor deposition of SiC onto a tungsten substrate. This fiber has a typical average tensile strength of 2700 to 3400 MPa and a tensile elastic modulus of approximately 415 GPa. The surface of the fiber is carbon rich as a result of the deposition process which includes the use of methane during the final stage of fiber formation. The carbon rich layer is responsible, in a major part, for the handleability and high tensile strength of the fiber.

The second fiber, obtained from the Nippon Carbon Company, is a highly flexible yarn, Fig. 1, which is fabricated by the conversion of an organometallic precursor. The surface of this fiber is extremely smooth, Fig. 2, in contrast to the normal "corn cob" character of fibers produced by CVD.

Fiber chemistry was analyzed using the Ion Scattering Spectrometer and Secondary Ion Mass Spectrometer. By irradiating the fibers with a primary beam of He<sup>3</sup> or Ne<sup>20</sup> it was possible to sputter ions from the fiber. These ions were analyzed in a mass spectrometer and readouts of ion current vs mass number were obtained for various locations within the fiber, Fig. 3. At a location of not more than 120A beneath the fiber surface in a beam of He<sup>3</sup> ions the presence of carbon (Atomic Mass Unit 12), silicon (AMU 28), and silicon carbide (AMU 40) was noted. Surprisingly, elements of sodium (AMU 23) and potassium (AMU 39) were also detected and are probably associated with some surface adherents. Similarly, the presence of peaks at AMU 12-15, 24-28 and 39-41 probably indicate the presence of organic material, and possibly some aluminum (AMU 27). The analysis at a depth of greater than 450A in a beam of Ne<sup>20</sup> revealed similar effects with equal peak heights of carbon, silicon and silicon carbide probably being characteristic of the bulk fiber. Any trace elements on the fiber surface would still appear in this analysis because, when analyzing a bundle of fibers, even though some sputtering has taken place, some original fiber surface will be left to contact the impinging beam of ions.

Quantitative chemical analysis of the entire fiber (not just the surface) has determined that, on an atomic scale, there is 22% excess carbon present. This would also tend to agree with the relatively low density of 2.89 gm/cm<sup>3</sup> as compared to 3.2 for pure SiC.



The tensile strengths of individual filaments from two shipments of fiber were determined. The first delivery, Set A, consisted of highly inflexible fibers of approximately  $40\mu$  in diameter. Their strengths are presented in Fig. 4 and typical fracture surfaces, as seen by use of the Scanning Electron Microscope, are shown in Fig. 5. Even for this large diameter, which is not considered optimum by the manufacturer, the average fiber strength is 1.6 GPa. This compares favorably with all of the fibers evaluated thus far on the program, and because of the lower density of SiC, as compared to fiber FP indicates considerable potential for the fiber as a reinforcement. The fiber fracture surfaces, Fig. 5, indicate failures due to both surface and internal defects.

The second set of fibers, Set B, was smaller in diameter,  $12\mu$ , and also higher in strength, Fig. 6. The average strength of 2.0 GPa is considerably higher than that of the larger diameter fiber Set A, and in addition this superior fiber is much more flexible. The yarn shown twisted in Fig. 1 is taken from this set of fiber. The fracture surfaces, Fig. 7, for the highest strength fibers indicate a dependence on surface flaws.

The data in Fig. 8 summarize the dependence of fiber strength on diameter. The range of data is that supplied by the manufacturer while the two data points refer to the above discussed UTRC data. It is seen that there is still considerable latitude for fiber strength improvement, even at the  $12\mu$  diameter currently available.

As the program progressed, a surface treated form of SiC yarn became available. The surface treated fiber has been shown to exhibit essentially the same mechanical properties as the original untreated fiber; however, it has a bluish color while the untreated fiber is black. A similar blue surface finish was achieved at UTRC by the oxidation of the untreated fiber at  $800^{\circ}\text{C}$  for several hours. It was the manufacturer's contention that the treated fiber should be more compatible with aluminum than the carbon rich untreated fiber.

Additional fiber mechanical property assessment was performed by the formation of SiC yarn reinforced epoxy matrix composites. By this procedure it was possible to determine the fiber effective bundle strength and elastic modulus. The data, Table I, indicate an elastic tensile modulus of approximately 220 GPa and a bundle strength of 1710 MPa.

Additional experiments were performed to determine the oxidative stability of the SiC yarn by exposure to air for a period of 1 hr at temperatures up to  $1100^{\circ}\text{C}$ . The data, Table II, indicate a measurable decrease in strength for exposure temperatures of as low as  $400^{\circ}\text{C}$  for both forms of fiber.

### III. FABRICATION AND PROPERTIES OF SiC YARN REINFORCED ALUMINUM COMPOSITES

The low strength and relatively small diameter of the SiC yarn precludes the effective use of any high pressure solid state powder metallurgical fabrication techniques. Extensive fiber fracture would take place due to both fiber-fiber and fiber-matrix powder contact during composite consolidation. By consolidating the matrix above the alloy solidus, however, it is possible to avoid fiber fracture. Two methods to achieve this end were used; liquid phase hot pressing and liquid metal infiltration.

#### A. Liquid Phase Hot Pressing

In this process, -325 mesh metal powder was infiltrated into the bundles of SiC yarn by the use of a slurry. The aluminum powder was first mixed with a carrier liquid of isopropyl alcohol. The fibers were then immersed in this agitated slurry, removed and allowed to dry. At that time the SiC fiber bundles contained approximately 8 to 10 times their weight in aluminum. Since both the yarn and matrix are nearly of the same density, this corresponded to an effective volume percentage of nearly 90% aluminum. The dried fiber plus powder bundles were then placed in a graphite die set, which in turn was placed inside a vacuum furnace. The hot pressing procedure involved pre-evacuation of the chamber to a level of approximately  $10^{-5}$  Torr followed by heating to the appropriate temperature and hot pressing. The chosen temperatures always exceeded the alloy solidus and usually also exceeded the liquidus temperature. (The aluminum alloys used in this program, and their melting temperature ranges, are listed in Table III.) As a result of this hot pressing operation most of the aluminum is extruded from the die leaving a higher percentage of fiber than originally inserted in the die.

A summary of the most significant composites fabricated is given in Table IV. The data can be divided into two groups; those composites fabricated with an applied pressure of 0.34 MPa, and those fabricated at 1.38 MPa. The former set of composites fabricated at low pressure were incompletely consolidated in all cases. In particular, the intra fiber tow spaces were incompletely filled with aluminum which resulted in excessive porosity and very poor bonding. This was true for both alloys used and also both forms of fiber. By increasing the hot pressing pressure, however, it was found that composites with nearly full consolidation were achievable at an applied pressure of 1.38 MPa. At this pressure most of the composite microstructure was pore free and fiber concentrations of up to 30% were achieved without fiber breakage. This latter point was ascertained by examining the condition of fibers removed from the densified composites by dissolution of the matrix in dilute hydrochloric acid.

The microstructure of a typical Al-Si alloy reinforced with approximately 30% SiC yarn is shown in Fig. 9. Several important features of this structure can be noted. First, there is a considerable range in fiber diameter evident, which is in agreement with our measurements of as received fibers. Secondly, the fibers are fairly uniformly distributed throughout the composite with matrix material completely surrounding each fiber. Finally, there is a considerable distribution of silicon particles which can be seen at higher magnification in Fig. 10. These particles consist of large primary particles and smaller particles formed during solidification of the eutectic. Many of these particles appear to be anchored on the SiC fibers indicating the likely role of the fiber surface as a preferred site for nucleation and growth from the melt. Similarly, the microstructure of 6061 matrix composites, Fig. 11, contained large numbers of particles that are probably  $Mg_2Si$ , and included oxides remaining from the original powders. In general the microstructures of the 6061 matrix composites reflected a greater tendency for "agglomeration" of the SiC fibers and a larger percentage of internal porosity.

A comparison between the data in Table IV and the data in Table I for SiC reinforced epoxy specimens indicates a definite inferior transfer of fiber strength to the metal matrix composites. An effective fiber bundle strength (in bending) of 1100 MPa is obtained for the composites in Table IV while a value of 1700 MPa was obtained from the epoxy matrix composites. This inferior strength transfer may be related to the mechanism of crack propagation in the composite, as well as possible fiber-matrix reaction. Crack growth in the metal matrix composite is discussed again later in this report when impact properties are examined.

The fracture morphology of the yarn reinforced aluminum specimens was examined by scanning electron microscopy, Figs. 12-14. The fracture surfaces consisted primarily of fibers which failed near or on the primary composite fracture plane. Fiber pull out of substantial length (greater than several fiber diameters in length) occurred infrequently and only in areas where there appeared to be a region of poor fiber-matrix bonding, Fig. 12. The fiber surfaces then appeared to be totally unaffected by their incorporation into the aluminum matrix. The other fracture surface areas, where little or no fiber pull out was observed, could be divided into two morphological types. The first, shown in Fig. 13, consisted of evidence for local interfiber ductile matrix rupture and high strength fiber-matrix bonding. The second, which was much less prevalent, Fig. 14, consisted of small regions in which the fibers were included within a brittle second phase particle.



B. Liquid Metal Infiltration

The liquid metal infiltration procedure, developed in the earlier phases of this program for alumina fiber reinforced aluminum, was far less successful than the previously described powder metal approach. Nonuniform metal distribution and incomplete infiltration characterized all of the composite rods fabricated. A typical as cast microstructure, shown in Fig. 15, was obtained using an Al-1.5% Li-7% Mg alloy. Because of these inferior results, the use of this procedure was discontinued.

#### IV. FABRICATION AND PROPERTIES OF SiC MONOFILAMENT REINFORCED ALUMINUM COMPOSITES

Unlike the SiC yarn discussed in the previous section, the SiC monofilament is highly resistant to damage by hot pressing in metal powder. Because of its 140 $\mu$  diameter size, it can be collimated and used to make precursor monolayer tapes which can then be stacked and processed to achieve bulk composite structures. This type of procedure has been previously developed for boron fiber reinforced aluminum (Ref. 2); however, because of the high tendency for fiber-matrix reaction in that system, there were severe processing limitations. Most significant of these was the inability to process composites in the presence of a liquid metal phase. Excessive reaction caused rapid loss of boron fiber strength. The SiC fiber offers potential for overcoming this limitation.

In each of the following processing procedures to be described, a precursor fiber plus matrix tape was fabricated. This tape is made by winding fiber, at a prescribed spacing, on a circular mandrel which has been previously wrapped with an aluminum foil. The foil is 25 $\mu$  thick, and can be one of a wide range of aluminum alloy compositions. A binder must then be applied over this foil plus fiber array to hold the fibers in position after removal of the tape from the mandrel. This binder can later form part of the final composite, or be a "fugitive" binder which is removed during the bulk composite consolidation step. In the former case the binder is an aluminum alloy which is applied to the tape by the plasma spraying of aluminum powder, while in the later case it is an organic material, such as polystyrene, which can be removed completely by heating under vacuum. In both cases, however, the monolayer tape can be cut into pieces which are then stacked for final bonding.

##### A. Solid State Diffusion Bonding

In the solid state diffusion bonding process, the temperature for composite consolidation is held to below the solidus of the aluminum alloy being used. Thus high pressures must be applied to achieve composite consolidation and full densification of the matrix. Also, to minimize oxidation of the matrix and fibers, the process is carried out in vacuum. In the past it has been usual for such a vacuum diffusion bonding process to take several hours with most of this time being consumed in the duration of system heat up and cool down. Because of these time requirements, this has been a very costly procedure and, even in the case of high material cost systems like boron reinforced aluminum, the fabrication expense has become the major contributor to final component cost. To overcome this cost problem, a process was developed for boron aluminum



at United Technologies which minimizes the time required in a hot press to achieve consolidation. Called "Quick Bond", (Refs. 3-5) this process utilizes preheated hot press platens located within a vacuum chamber and can be described by the following steps.

- (1) Preheat platens to anticipated hot pressing temperature. The vacuum chamber is now filled with air.
- (2) Suspend the multi-ply composite tape lay-up between the platens so that it is not in direct contact with either platen.
- (3) Seal off the vacuum chamber and pump down the system to a level of better than  $10^{-3}$  Torr. Using a high capacity pumping system, steps 1 through 3 can be completed in 10 minutes or less.
- (4) Bring the platens together to cause contact and rapid heating of the composite. After the composite has reached the desired temperature, apply full pressure to consolidate the composite.
- (5) Release the vacuum, separate the platens, and remove the diffusion bonded composite.

The entire process can be carried out in less than 30 minutes for even large composite panels, and represents a major increase in efficiency over the more common multi-hour processes practiced throughout the composite industry.

SiC monofilament reinforced composites were fabricated by this process using four different aluminum alloy systems, Table V. In each case a plasma sprayed tape was first made using the foil and powder compositions listed. The alloys chosen are representative of the generally important classes of aluminum alloys used in engineering applications, i.e. 713/528 (Al-Si alloys), 6061/6061 (Al-Mg-Si alloys), 2024/2024 (Al-Cu-Mg alloys) and 5052/5056 (Al-Mg alloys), Table III. The hot pressing procedures listed in Table V are: the temperature at which diffusion bonding took place (the maximum temperature of the composite under full pressure), the maximum pressure applied and the time over which this maximum pressure was applied. Although other hot pressing conditions were tried, those listed in the table provided the best composite microstructures and performance. In each case the 550°C temperature approaches, to within approximately 25°C, the alloy solidus. Only in the case of the 2024 alloy was the solidus exceeded, and in this instance the hot pressing pressure was lowered substantially.

In each of the cases given in the table it was possible to achieve a dense, well bonded composite. The polished and etched microstructure of a SiC reinforced 2024 composite, Fig. 16, indicates that the aluminum matrix flowed readily around the fibers and bonded the entire powder plus foil microstructure together.

Although this full densification was the case, it did not insure good bonding between the fiber and the matrix. As had been discussed previously, the surface of the SiC monofilament is fabricated carbon rich, and this condition inhibits bonding to aluminum. Thus, as shown in Fig. 17, the fracture surface of axial tensile specimens is characterized by substantial fiber pull out and no evidence of fiber-matrix reaction. This observation is independent of solid state bonding condition and aluminum alloy composition. It is interesting to note that nearly all of the fiber fracture surfaces indicate that fiber fracture initiated near the core of the fiber, and not at the outer surface. This also is in agreement with the observed lack of fiber-matrix interaction. Upon closer inspection of the fiber fractures, Fig. 18, it can also be seen that the fiber core has a duplex structure. The manufacturer has indicated that the core consists of an approximately  $35\mu$  diameter graphite filament and, as shown in the figure, this core has a thin 1 to  $2\mu$  surface coating or reacted interface separating it from the SiC fiber body. Thus, during composite fracture, not only is there fiber pull out from the matrix, but there is also fiber core pull out from the overall fiber.

Because of the higher as-received strength of the SiC monofilament, and the apparent lack of fiber-matrix reaction, the axial strengths of the composites given in Table V are considerably higher than those for either of the other systems tested during this program, i.e. the FP reinforced aluminum or SiC yarn reinforced aluminum. The highest strengths shown are approximately 10-20% less than those obtained with boron fiber reinforced aluminum composites of the same matrix type and this strength differential can be ascribed to the lower strength of the SiC filament.

Composite transverse strength is also affected by the fiber-matrix bond condition. In general, it has been found that the transverse tensile strength of metal matrix composites can equal the tensile strength of the unreinforced matrix, (Ref. 6). This situation prevails, however, only if the composite fractures through the matrix. Thus, the relatively low values of transverse strength presented in Table V are due to composite failure through the fiber-matrix interface. This is again due to the poor fiber-matrix bond. The highest transverse strength was achieved for the 2024/2024 matrix composite which was bonded at a temperature exceeding the matrix solidus which may have promoted some fiber-matrix reaction and bonding.

The above discussed composites were all fabricated using a vacuum atmosphere. For the low cost fabrication of large composite panels, however, it may be desirable to fabricate composites in a less demanding atmosphere. Previous work at UTRC has shown that the plasma sprayed aluminum coating on a boron reinforced aluminum tape, protects the boron fibers from rapid oxidation in air for at least short periods of time (Ref. 3). Therefore, since the SiC fiber is more

stable in an oxidizing environment than boron, it was anticipated that this lower cost alteration in atmosphere should not pose a problem. The composites listed in Table VI were thus fabricated in air. The procedure differs from the previously described vacuum bonding procedure only in that the composite is not initially suspended between the platens and there is no vacuum pump down. Instead, the composite is immediately sandwiched between the preheated platens to maximize the rate of composite temperature rise. After attainment of the bonding temperature the process proceeds just as in the previous vacuum bonding procedure, however, in this case in air.

The data presented in Table VI emphasize only the transverse composite strength since it is this quantity which would be most affected by exposure to air. Extensive preoxidation of the aluminum could interfere with composite bonding. Additional alterations in procedure included the use of higher bonding pressures, which definitely improved the transverse strength of the 6061 matrix composites, and increasing the bonding temperature to just above the solidus for each alloy. Unfortunately, in all cases the transverse composite strength remained much lower than that of the monolithic matrix and all fracture surfaces, as in the case of vacuum bonding, were characterized by fiber-matrix debonding.

#### B. Liquid Phase Diffusion Bonding

Another procedural modification which can lower composite fabrication cost is the use of low pressure during composite consolidation. In this way large panels can be made without the need for extremely large hot press load capacity. This decrease in pressure can only be accomplished, however, through the elevation of the bonding temperature to above the metal solidus. To this end, a number of composites were processed in the "Quick Bond" mode under vacuum using a platen applied pressure of approximately  $2\frac{1}{2}$  atmospheres. These experiments were performed using Al-Si alloys because of the superior cast structures possible using brazing filler alloys. The 718, aluminum-silicon eutectic composition, alloy was used with a fugitive tape binder of polystyrene, since a eutectic powder was not available, while the 713/528 alloy composition was used in plasma sprayed tape form. The data presented in Table VII represent the best conditions observed to achieve full consolidation of the composite. Unfortunately, these conditions also appeared to cause degradation of the composite axial tensile strength. It was observed that fibers extracted from these composites were quite brittle and weak, undoubtedly due to excessive fiber-matrix reaction.



Another approach to overcome this problem is to preconsolidate a composite and then, without melting the matrix which surrounds the fibers, braze bond together a thicker composite. This was accomplished by first preconsolidating SiC/6061 bi-layer composite panels using the solid state diffusion bonding approach. These composite pieces were then brazed together in vacuum using a "Quick Bond" procedure, Table VIII. The resultant composite strength was well above that achievable by the previous all liquid phase processing. The brazing temperature used exceeded the solidus of the 6061 matrix slightly; however, very little melting of the 6061 took place, in light of the much higher liquidus temperature of 650°C.

## V. COMPOSITE IMPACT PROPERTIES

One of the major concerns, in the use of composite materials, is their ability to resist impacts and tolerate damage. Previous work by many investigators has demonstrated that the ability to dissipate impact energy in metal matrix composites is a strong function of fiber diameter and matrix composition. SiC monofilament, SiC yarn, and FP alumina yarn Charpy impact specimens were tested to compare these systems. In each case the specimens were V-notched prior to test and all testing was performed on an instrumented impact tester. The instrumentation was provided by strain gages located on the striker tup so that a load-time trace for each specimen could be obtained. In all cases except one the specimens were full sized Charpy V-notch dimensions. The exception was for the SiC yarn reinforced material which was only 65% of the thickness (direction perpendicular to crack growth) of a full sized specimen.

The data, Table IX, reveal a major difference in performance between the three composite systems. Both in energy dissipation capability and maximum load prior to failure, the large diameter SiC fiber reinforced material is superior. This can be associated with the major variations in composite tensile strength, as described previously in this report, and the considerable variation in fiber-matrix interaction at the fracture plane. Scanning electron microscopic examinations of the fracture surfaces clearly point out these differences. The large diameter SiC fiber protrudes over long lengths from the fracture surface, Fig. 19. This has caused extensive energy dissipation through crack diversion and fiber pull out. In contrast, both the FP and SiC yarn reinforced specimens fractured with almost no evidence of fiber-matrix separation, Figs. 20,21. The FP reinforced material exhibited the least amount of fiber extension above the fracture plane due to the three factors; small fiber diameter, relatively low fiber strength, high matrix-fiber bond strength. All three of these factors tend to reduce fiber pull out. Even though the matrix was still capable of deforming in a ductile manner around each fiber, very little energy was dissipated during crack propagation. Only at one small location on the fracture surface was the crack diverted by pull out and this was due to a defect which resulted in incomplete fiber-matrix bonding. The SiC yarn reinforced aluminum similarly, because of the low fiber strength and small fiber diameter, caused almost no diversion to advancing crack growth.



## VI. CONCLUSIONS

Although this research program was first begun with the intent of investigating the fabricability and properties of only alumina fiber reinforced aluminum matrix composites, its major objective of developing new, potentially lower cost, metal matrix composites warranted the inclusion of two types of SiC fibers. Both fibers exhibit substantial improvements in fiber specific strength over alumina fibers due to their lower density and higher strength; however, the translation of this strength into composite form was found to be difficult because of the carbon rich surfaces of both fibers. As in the case of graphite fiber reinforced aluminum, which in past years has been the subject of extensive research, the casting of aluminum alloys around the bare SiC fibers was unsuccessful. However, by resorting to a powder metallurgical approach, it was possible to achieve fully dense composites for both fibers.

The consolidation of metal powder plus fiber preforms was not found to be as sensitive to the existent matrix-fiber incompatibility and, as a result, it was even possible to raise the bonding temperature to above the aluminum alloy solidus and still achieve dense, high strength composites. In the case of the large diameter SiC monofilament, the composite axial strength values were the highest of any obtained during the entire program, and compare favorably with those of the well developed boron reinforced aluminum system. In fact, in all aspects, except transverse tensile strength, it is expected that the large diameter SiC fiber reinforced aluminum system can compare favorably with boron reinforced aluminum.

The SiC monofilament reinforced aluminum composite also exhibited superior impact characteristics, when compared to both the alumina and SiC yarn reinforced alloys. This is of considerable importance to metal matrix composite applications and points out a major difficulty. Small diameter fiber yarns have the greatest potential for future low cost because they can take advantage of the technology base already well developed in the textile industry for high volume fiber production. Unfortunately, however, the toughness and impact resistance of composites are adversely affected by small fiber diameter, particularly if the fiber-matrix bond is a strong one. Thus, both the FP alumina and SiC yarn reinforced composites are unable to effectively divert crack growth across the principal fiber direction. Although not demonstrated in the current program, this could also imply low fatigue crack growth resistance and an undesirable sensitivity to machining and fabrication induced defects. This should not, however, be a problem for the monofilament SiC reinforced composite which, as for B/Al, should exhibit exceptional fatigue crack growth resistance and fracture toughness. In addition, it is currently projected\* that the SiC monofilament will cost far less (up to 50% less) than boron fiber to produce.

\*Avco Corporation cost projections based on equal volumes of boron and silicon carbide fibers produced.

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5. Delgrosso, E. J., et al: "Development of Air Bonded FOD Resistant Metal Matrix Fan Blades", AFML-TR-77-13.
6. Prewo, K. M. and K. G. Kreider: "High Strength Boron and Borsic Fiber Reinforced Aluminum Composites", J. Comp. Mats., Vol. 6, p 338, 1972.
7. McDanel, D. L. and A. Signorelli: "Effect of Fiber Diameter and Matrix Alloys on Impact-Resistant Boron/Aluminum Composites", NASA-TN-D8204, 1974.
8. Prewo, K. M.: "The Charpy Impact Energy of Boron-Aluminum", J. Comp. Mats., Vol. 6, p 442, 1972.

Table I

Composite Tensile Properties for  
Nippon - SiC Reinforced Epoxy

<u>Fiber Content</u> v/o	<u>UTS</u>		<u>E</u>		$\frac{\epsilon_f}{\%}$
	MPa	$10^3$ psi	GPa	$10^6$ psi	
46	876	127	103	14.9	0.79
	771	112	108	15.7	0.67
	712	103	98	14.2	0.75
Avg.	786	114	103	14.9	0.74

Therefore the fiber properties are:

$$E_f = 221 \text{ GPa } (32 \times 10^6 \text{ psi})$$

$$\sigma_f = 1710 \text{ MPa } (250 \times 10^3 \text{ psi})$$

$$1630 \text{ MPa } (236 \times 10^3 \text{ psi}) \text{ based on } E \epsilon_f$$

$$\rho_f = 2.6 - 2.8 \text{ gm/cc}$$

Table II

Tensile Strength of SiC Yarn After  
1 Hr Thermal Exposure in Air

<u>Exposure Temp.</u> °C	<u>Unsurface Treated Fiber</u> UTS $\pm$ Std Dev (MPa)	<u>Surface Treated Fiber</u> UTS $\pm$ Std Dev (MPa)
Unexposed	1710 $\pm$ 530	1780 $\pm$ 70
400	1340 $\pm$ 540	1400 $\pm$ 580
800	1340 $\pm$ 530	1400 $\pm$ 590
1100	1075 $\pm$ 250	1200 $\pm$ 600

Table III

## Aluminum Alloys

<u>Designation</u>	<u>Composition</u> (wt %)	<u>Solidus</u> °C	<u>Liquidus</u> °C
6061	1 Mg, 0.6 Si, 0.25 Cu, 0.25 Cr	582	652
2024	4.5 Cu, 0.6 Mn, 1.5 Mg	502	638
4343	7.5 Si	577	613
713	7.5 Si	577	613
716	10 Si, 4 Cu	522	585
718	12 Si	577	582
LSA 528	7.5 Si	577	613
LSA 538	10 Si, 4 Cu	522	585
5052	2.5 Mg	593	649
5056	5.2 Mg	568	638



Table IV

SiC Yarn Reinforced Aluminum Composites Fabricated  
by Liquid Phase Hot Pressing

Comp. Number	Fiber	Matrix	Hot Pressing Conditions			Resultant Composite		
			Temp. °C	Pressure MPa	Time min	Density gm/cm <sup>3</sup>	v/o Fiber	Flexural Strength MPa
3049	Untreated	6061	650	0.34	15	2.44	Incomplete Densification	
3051	Untreated	6061	700	0.34	15	2.31		
3052	Surface Treated	6061	700	0.34	15	2.29		
3050	Untreated	LSA-538	592	0.34	15	2.2		
3053	Surface Treated	LSA-538	650	0.34	15	2.64		
3054	Surface Treated	LSA-538	700	0.34	15	2.0		
3062	Untreated	6061	700	1.38	15	2.47	30	306 322 346
3064	Surface Treated	6061	700	1.38	15	2.48	30	380 330 242 335
3060	Untreated	LSA-538	595	1.38	15	2.60	20	299 310
3061	Untreated	LSA-538	595	1.38	15	2.47	30	306 322 346
3063	Surface Treated	LSA-538	600	1.38	15	2.52	28	375 330 281
3065	Ni Plated	LSA-538	600	1.38	15	Incomplete Densification		

Table V  
SiC Monofilament Reinforced Aluminum Composites Fabricated by  
Diffusion Bonding in Vacuum

Comp.	Foil	Tape Binder	Hot Pressing Conditions		v/o Fiber	Resultant Composite Tensile Strength (MPa)	
			Temp °C	Pressure MPa		0°	90°
3019	713	528	500	34.5	51	1123 1130 1160	- - -
3020	713	528	550	34.5	52	1212 1384 1275	- - -
3022	6061	6061	500	34.5	48	1245 1390 1330	- - -
3021/25	6061	6061	550	34.5	48	1212 1495 1500	49.3 48.3 64.1
3043/44	5052	5056	500	34.5	48	1200 1124 1130	42.0 38.6 40.0
3041/42	5052	5056	550	34.5	48	1123 1206 1192	46.2 42.7 40.9
3045/46	2024	2024	490	34.5	51	1412 1336 1330	42.0 44.4 42.0
3047/48	2024	2024	540*	1.4	44	1120 1054 960	65.9 66.4 69.5

\*This temperature exceeds the alloy solidus and thus some liquid phase is present during consolidation.

Table VI  
SiC Monofilament Reinforced Aluminum Composites Fabricated  
by Diffusion Bonding in Air

Comp.	Foil	Tape Binder	Hot Pressing Conditions			v/o Fiber	Resultant Composite	
			Temp °C	Pressure MPa	Time min		Tensile Strength (MPa)	
							0°	90°
3077/93	2024	2024	490	68.9	15	49	1510 1460 1370 1480	38.2 64.5
3078	2024	2024	490	68.9	30	51		47.2 66.2
3079	2024	2024	500	34.5	30	49		66.8 62.1
3071	6061	6061	550	68.9	30	54		97.8 98.0
3072	6061	6061	570	68.9	30	52		75.0 105.0
3073	6061	6061	585	34.5	15	52		74.1 86.3
3074	713	528	550	68.9	30	51		60.5 55.0
3075	713	528	570*	68.9	30	51		81.4 84.0
3076	713	528	585*	34.5	15	55		53.1 41.2

\*Evidence of matrix melting

Table VII

SiC Monofilament Reinforced Aluminum Composites Fabricated by  
Liquid Phase Diffusion Bonding in Vacuum

<u>Comp.</u>	<u>Foil</u>	<u>Tape Binder</u>	<u>Hot Pressing Conditions</u>			<u>Resultant Composite</u>	
			<u>Temp.</u> °C	<u>Pressure</u> MPa	<u>Time</u> min	<u>v/o Fiber</u>	<u>0° Tensile Strength</u> MPa
2988	718	Polystyrene	595	0.24	7.5	46	655 580 800
2989			600	0.24	7.5	58	540 485 612
2990			595	0.24	12.5	54	500 444 504
2991			600	.10	7.5	44	703 800 695
2993	713	528	600	0.24	7.5	51	854 1000 792
2994			610	0.24	7.5	56	785 890 833



Table VIII

Braze Bonding of SiC Monofilament Reinforced 6061

- (1) Original 2-tape ply composites bonded together by solid stated vacuum diffusion bonding at 550°C, 34 MPa, 15 minutes
- (2) Secondary braze bonding accomplished using 713 Al at 595°C, 1.4 MPa, 15 minutes in vacuum

Resultant Composite Tensile Strength

1140 MPa  
1290  
1110  
1320  
1365  
1290

Avg. 1250 MPa

Table IX

## Instrumented Impact Test Comparison at 22°C

<u>Specimen</u>	<u>Material</u>		<u>Specimen Size</u>	<u>E*</u> Joules	<u>P<sub>max**</sub></u> N
	Fiber	Matrix			
3088	50% Avco SiC	6061	Full	21.8	5612
FP-1	60% FP	Al-Li	Full	0.43	-
FP-2	60% FP	Al-Li	Full	0.34	1962
3096	30% SiC yarn	Al-Si	Sub	0.20	1219
3096	30% SiC yarn	Al-Si	Full <sup>+</sup>	0.30 <sup>+</sup>	1852 <sup>+</sup>

<sup>+</sup> These values were obtained by increasing the data for subscale specimen 3096

\*Total energy dissipated during impact test (1 ft lb = 1.356 J)

\*\*The maximum load recorded by instrumentation prior to specimen fracture

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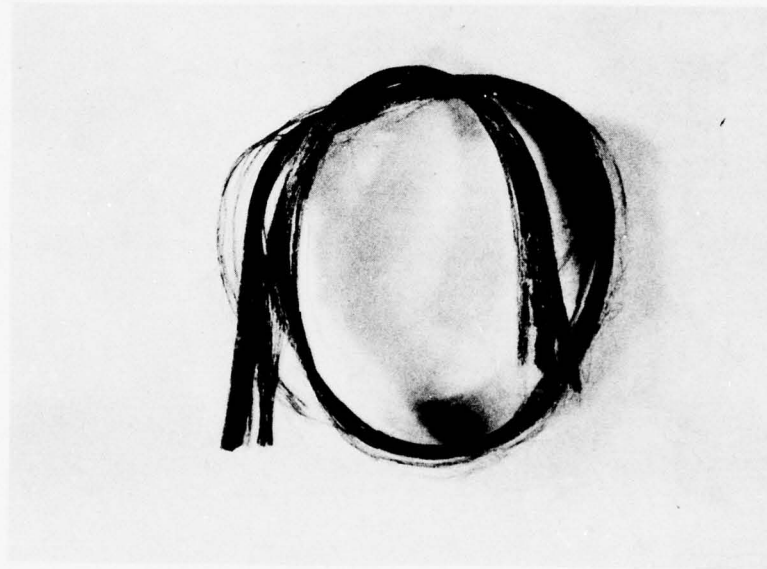


FIG 1. SiC YARN

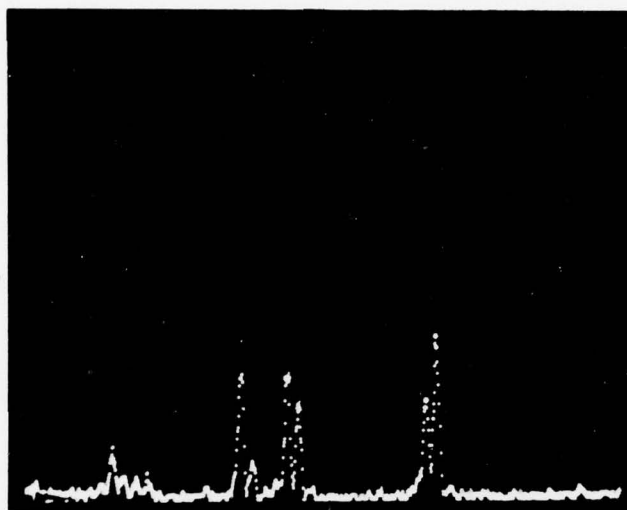
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FIG. 2 SURFACE OF SiC YARN FILAMENT



PEAK  
INTENSITY

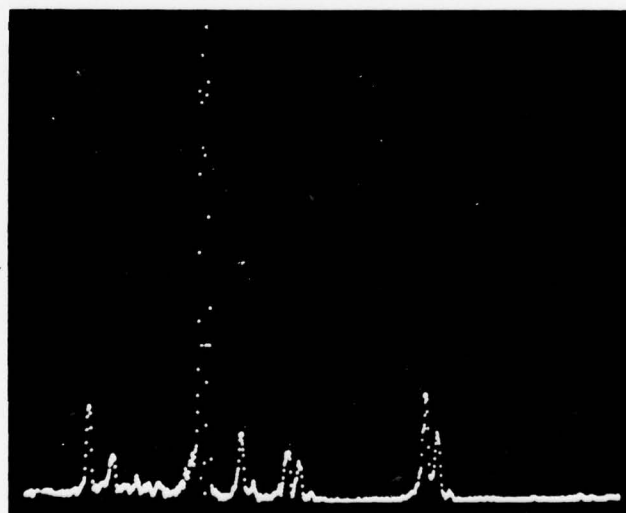


12 23 24 27 28 39 40

→ ATOMIC MASS UNITS

SPECTRUM TAKEN AT 0-120 Å BENEATH SURFACE

PEAK  
INTENSITY



10 12 20 23 27 28 39 40

→ ATOMIC MASS UNITS

SPECTRUM TAKEN AT 450-750 Å BENEATH SURFACE

FIG. 3 SIMS MASS SPECTRA OF SiC YARN

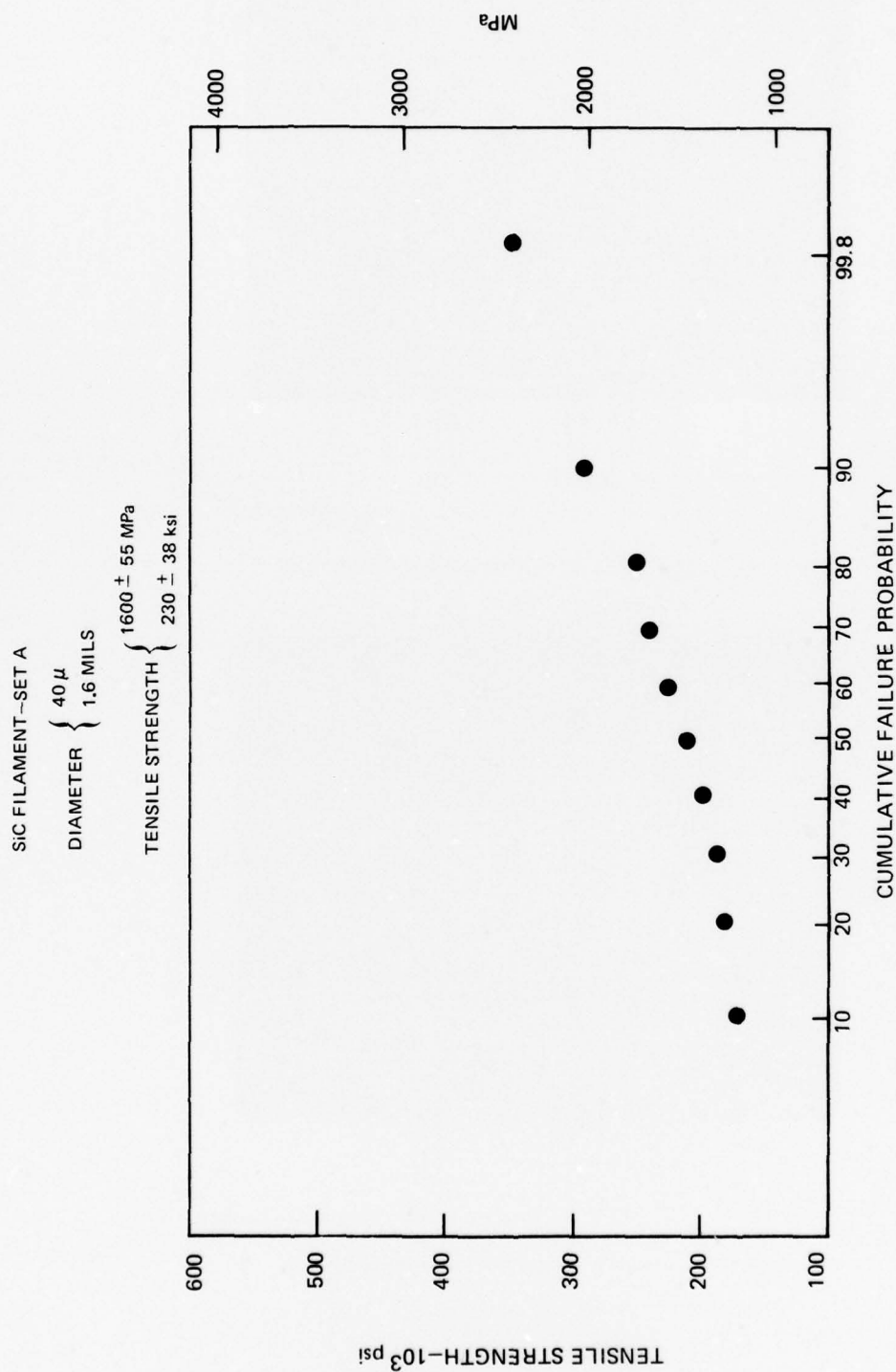
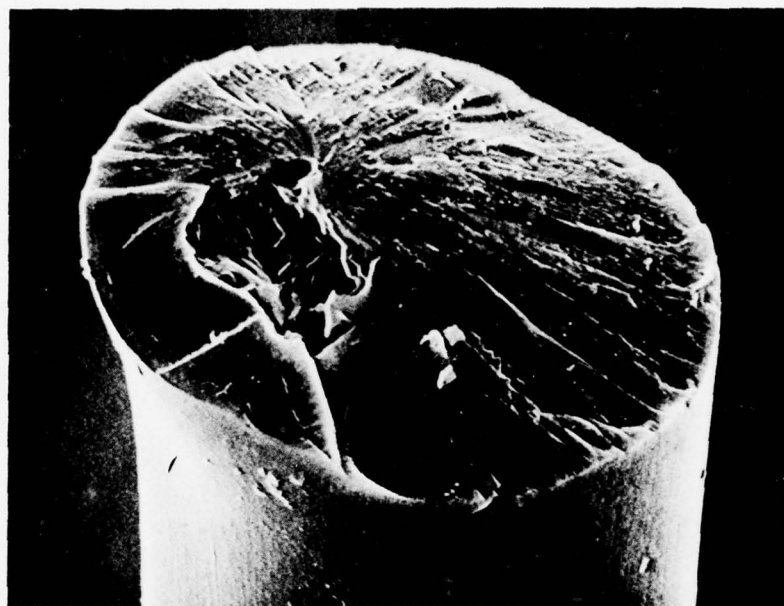


FIG. 4 TENSILE STRENGTH OF SIC YARN INDIVIDUAL FILAMENTS

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UTS = 1240 MPa (180,000 psi)



UTS = 2410 MPa (350,000 psi)

FIG. 5 SiC FILAMENT FRACTURE SURFACES — SET A

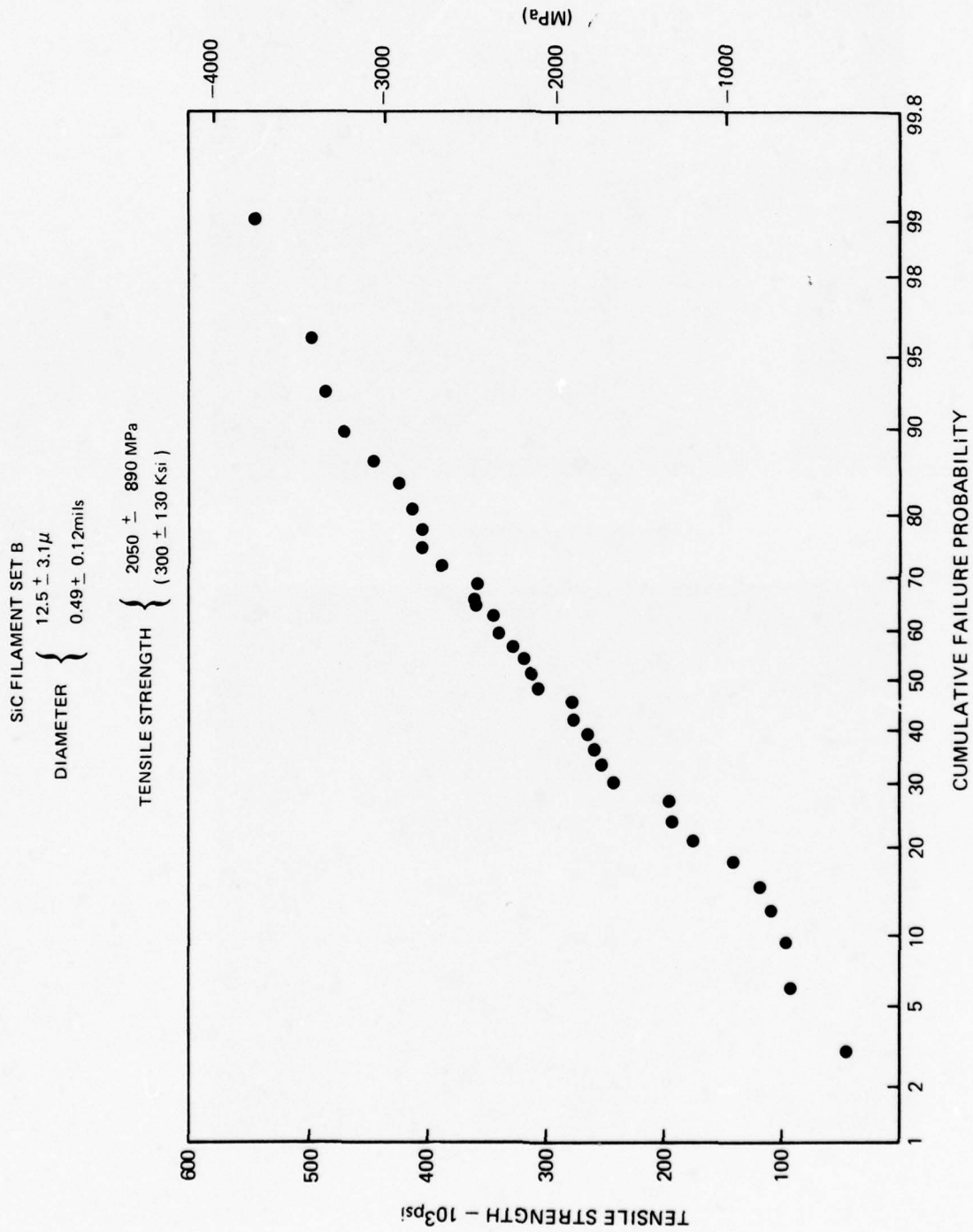
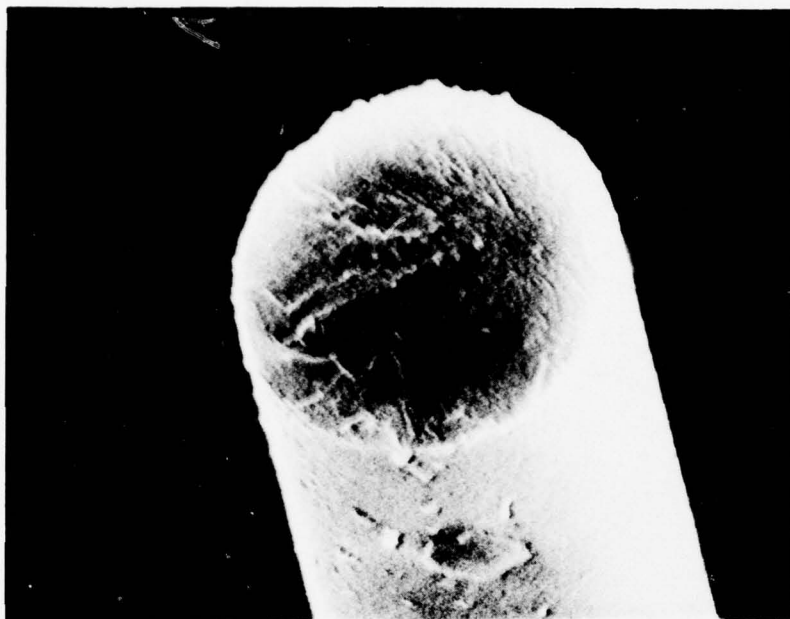


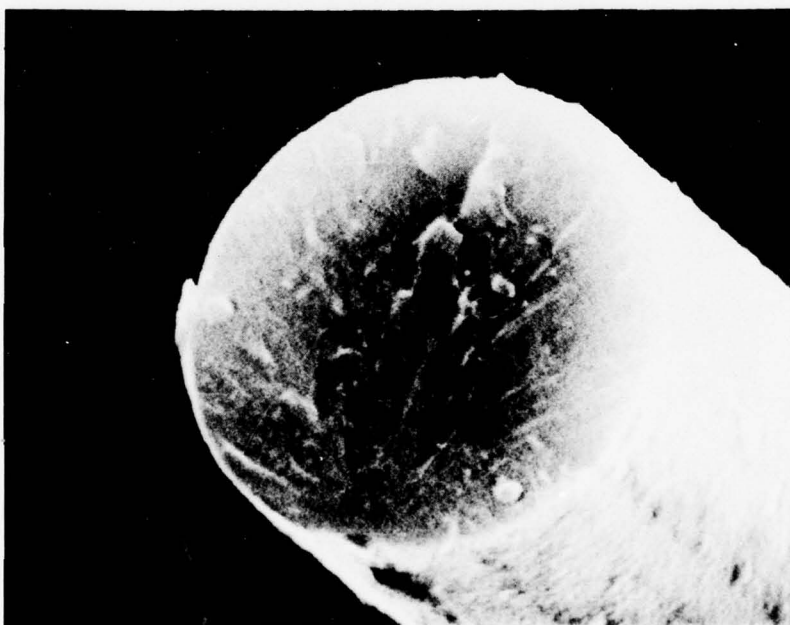
FIG. 6 TENSILE STRENGTH OF SiC YARN INDIVIDUAL FILAMENTS





UTS = 3330 MPa (480,000 psi)

2 $\mu$



UTS = 3400 MPa (495,000 psi)

1 $\mu$

FIG. 7 SiC FILAMENT FRACTURE SURFACES - SET B

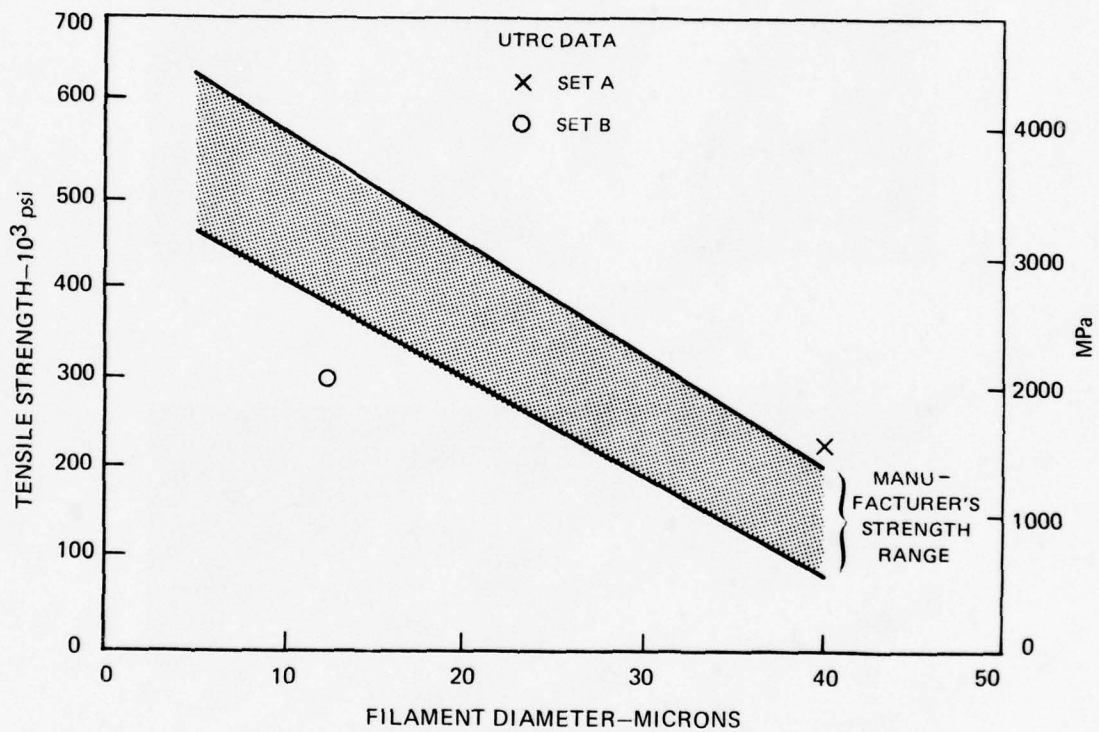


FIG. 8 SiC FILAMENT TENSILE STRENGTH

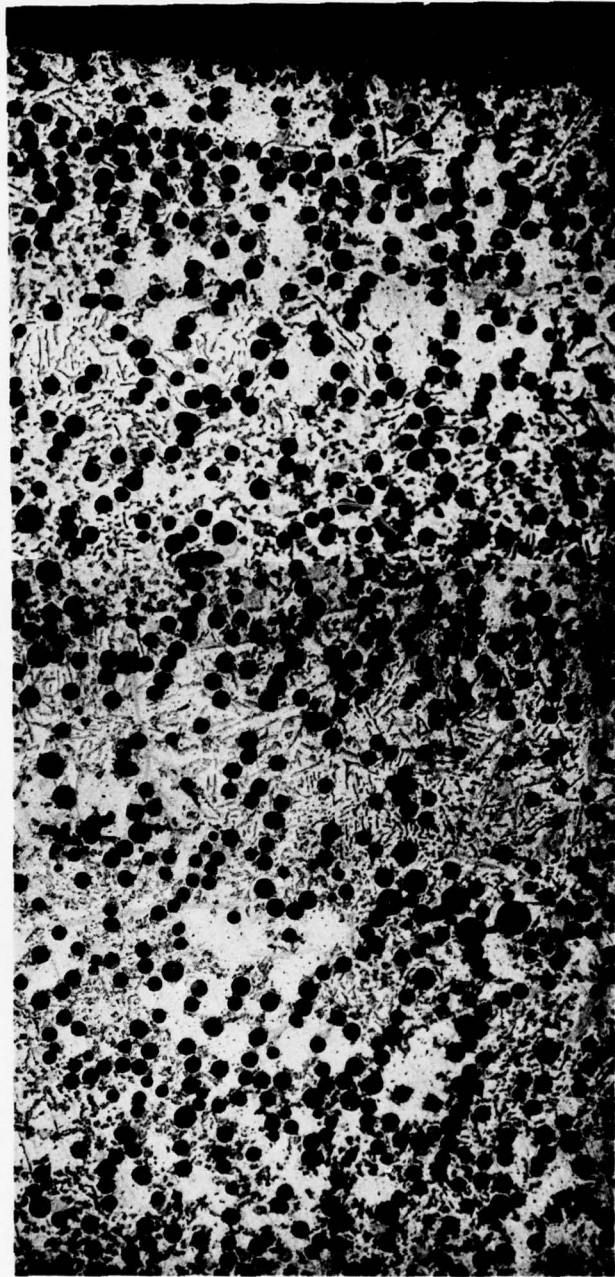


FIG. 9 TRANSVERSE SECTION THROUGH THICKNESS OF SPECIMEN 3063.  
SiC YARN REINFORCED LSA 538

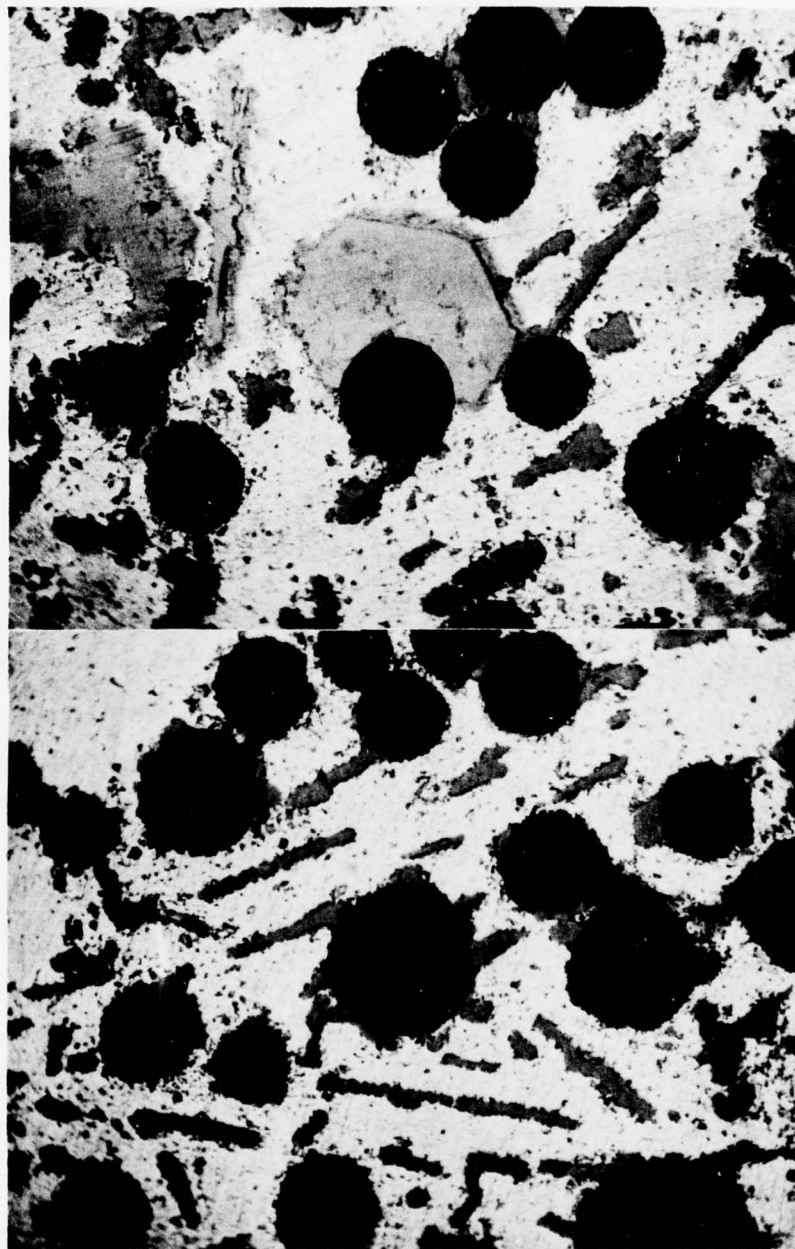


FIG. 10 MICROSTRUCTURE OF SiC YARN REINFORCED LSA 538 ALUMINUM



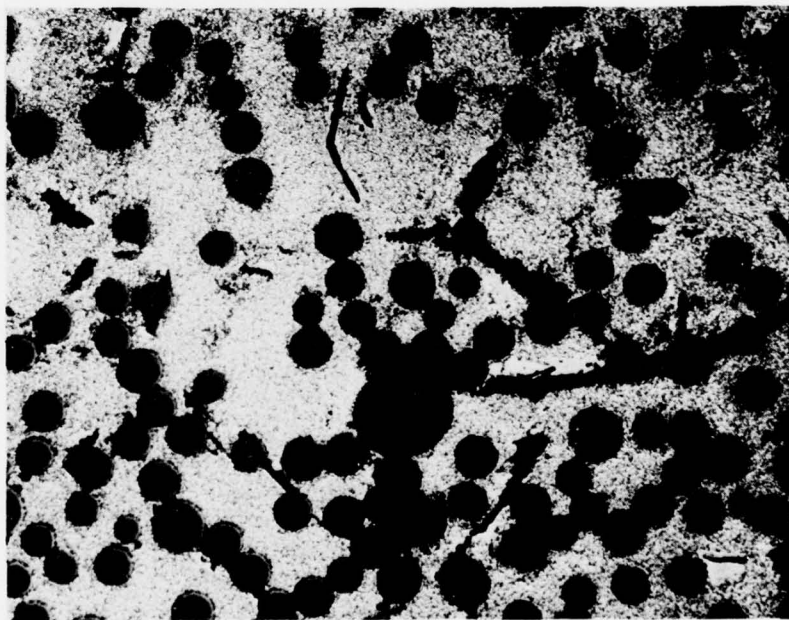
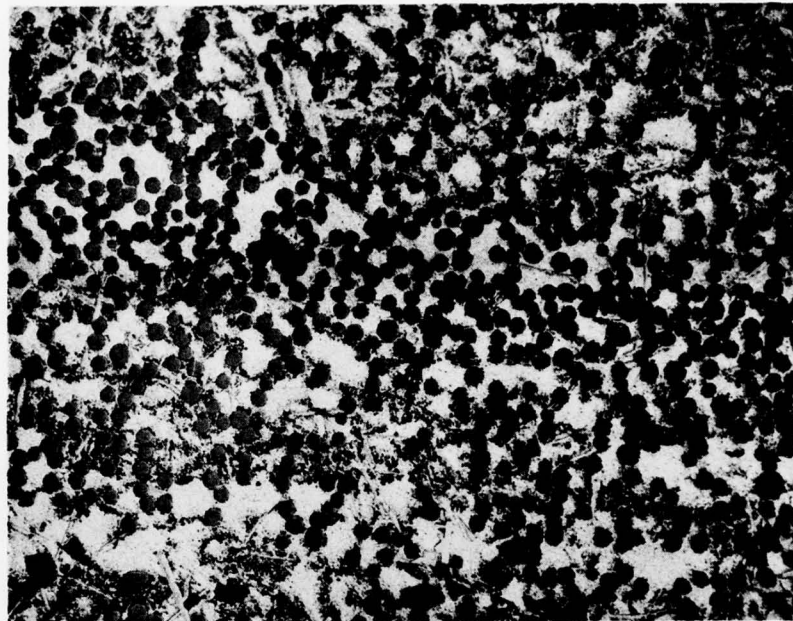
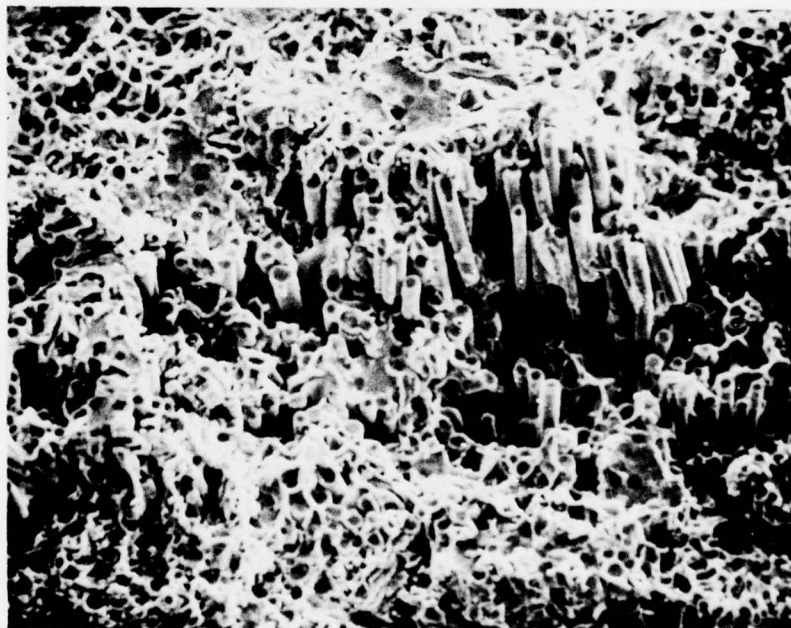


FIG. 11 MICROSTRUCTURE OF SiC YARN REINFORCED 6061 ALUMINUM

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400 $\mu$



50 $\mu$

FIG. 12 FRACTURE SURFACE OF SiC YARN REINFORCED 6061  
ALUMINUM SPECIMEN 3064

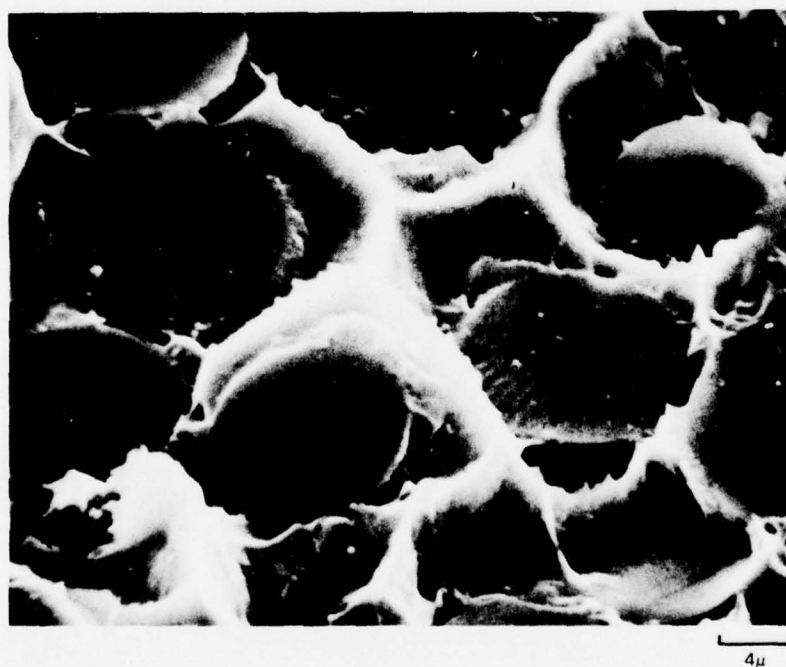
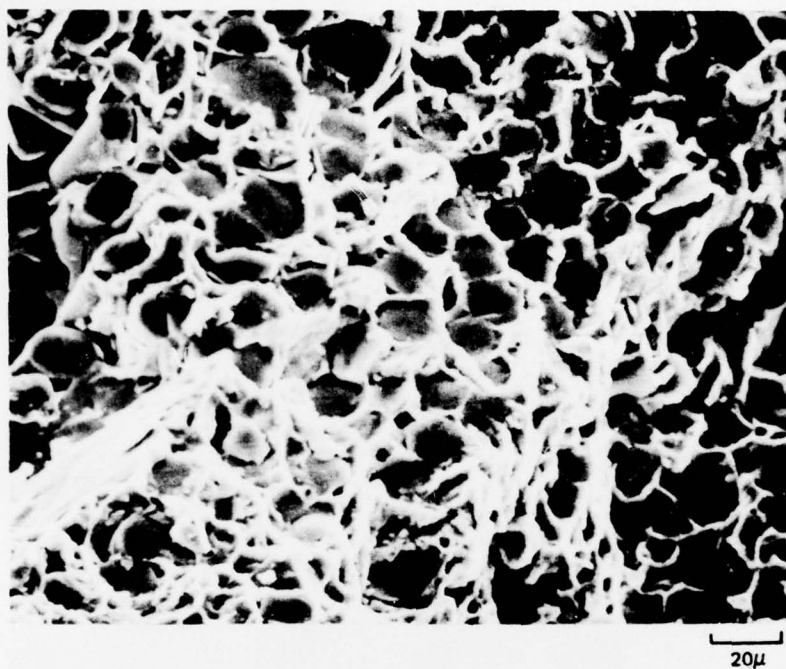


FIG. 13 FRACTURE SURFACE OF SiC YARN REINFORCED  
LSA 538 ALUMINUM SPECIMEN 3063

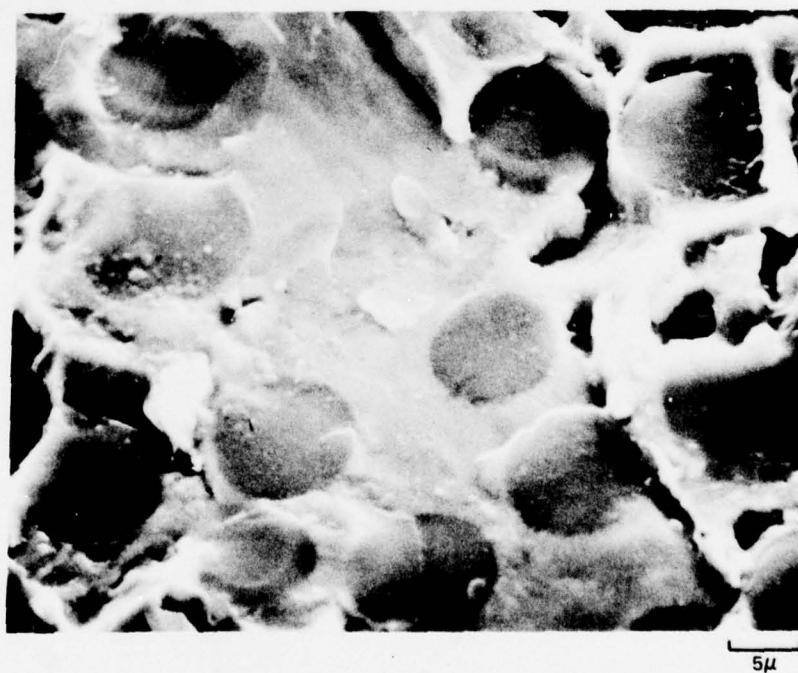
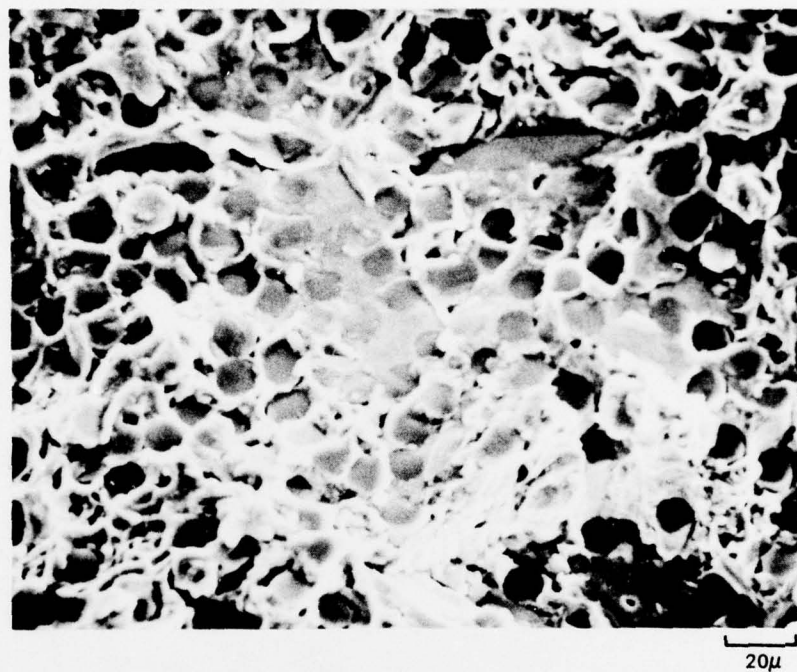


FIG. 14 FRACTURE SURFACE OF SiC YARN REINFORCED  
6061 ALUMINUM SPECIMEN 3064



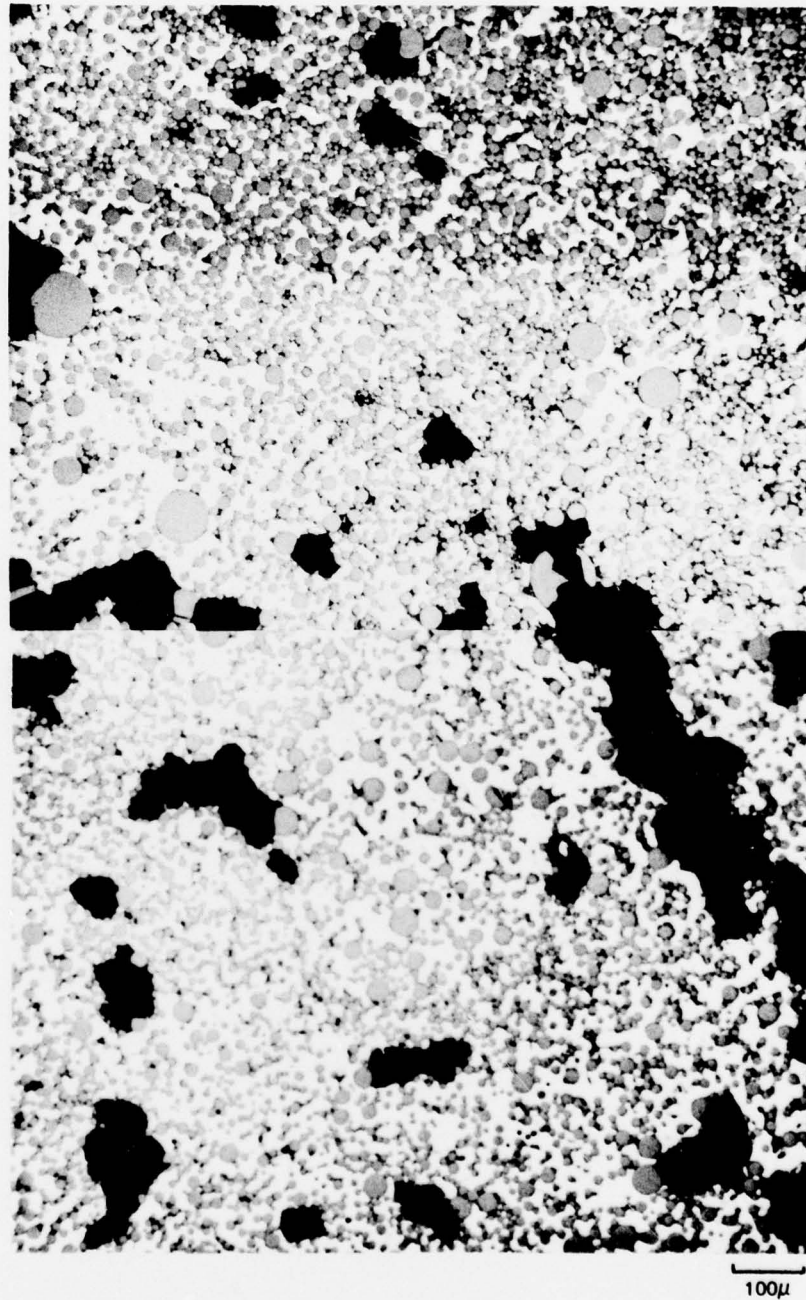
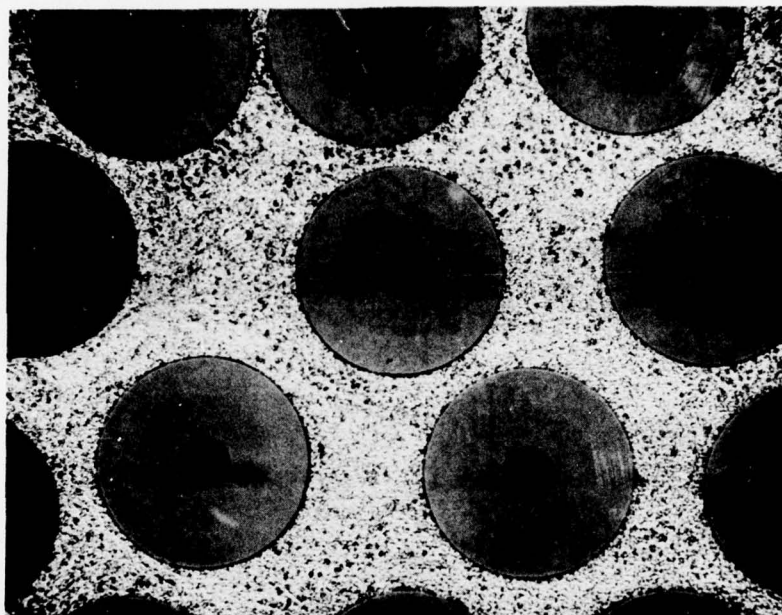
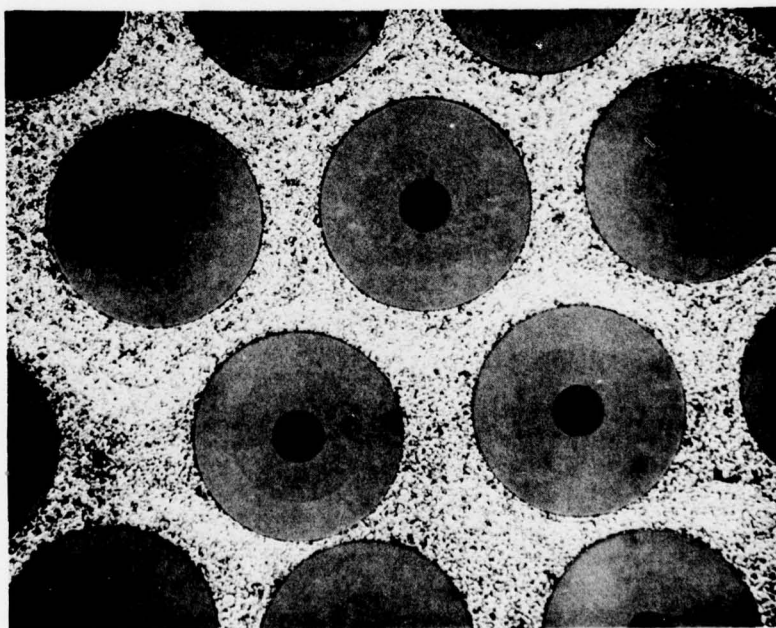


FIG. 15 SiC YARN IN Al-1.5% Li-7% Mg ALLOY FABRICATED BY LIQUID METAL INFILTRATION

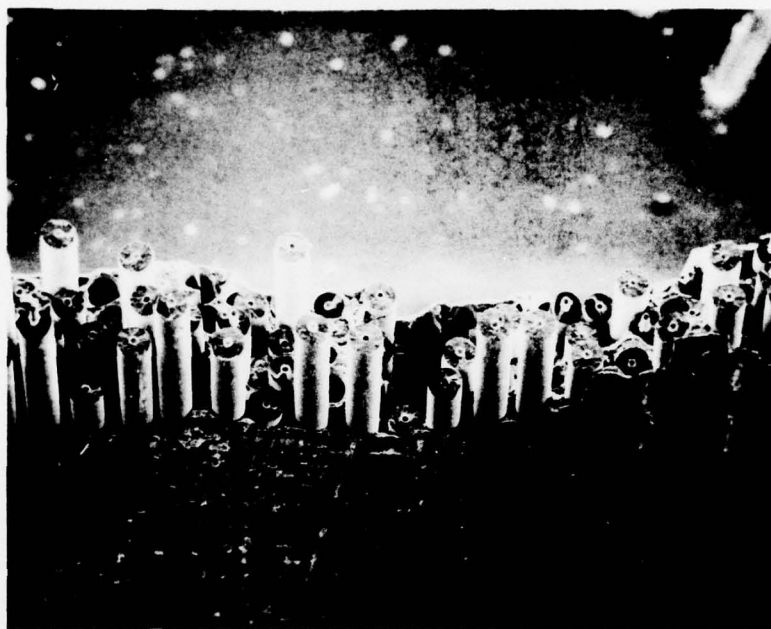


50μ

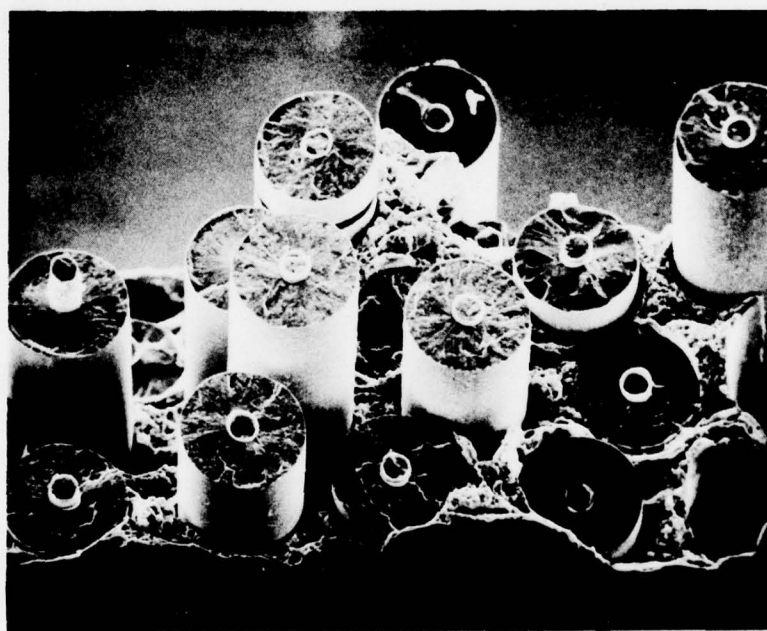


50μ

FIG. 16 MICROSTRUCTURE OF SiC MONOFILAMENT REINFORCED 2024/2024  
ALUMINUM - SPEC, 3045



400μ



100μ

FIG. 17 FRACTURE SURFACE OF AXIAL TENSILE SPECIMEN OF SiC MONOFILAMENT  
REINFORCED 2024/2024 SPEC 3045

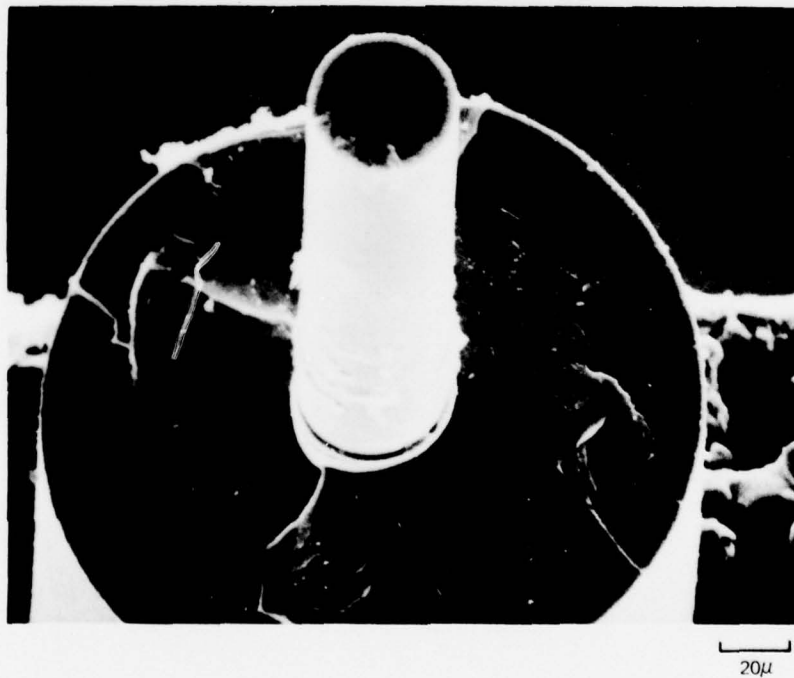
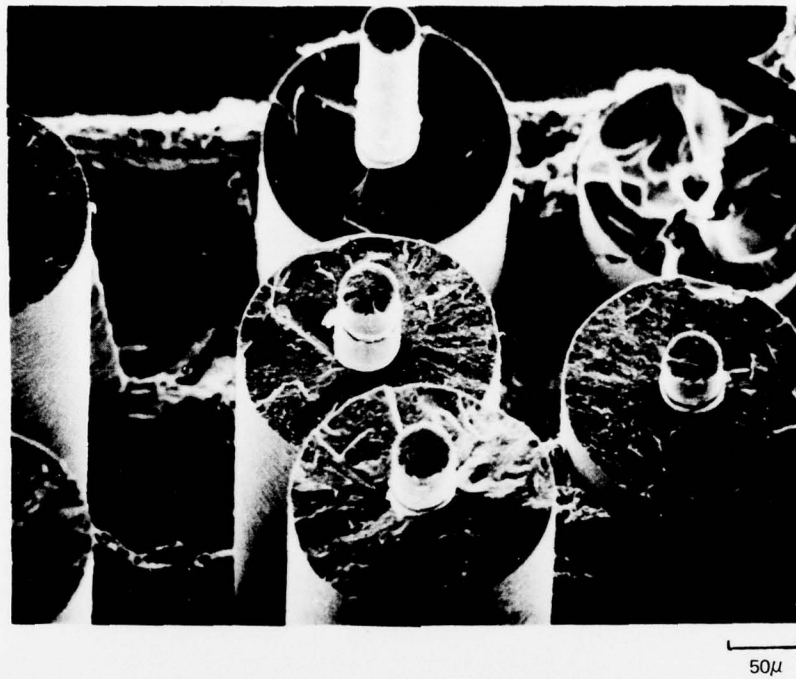


FIG.18 SiC MONOFILAMENT FRACTURE SURFACE



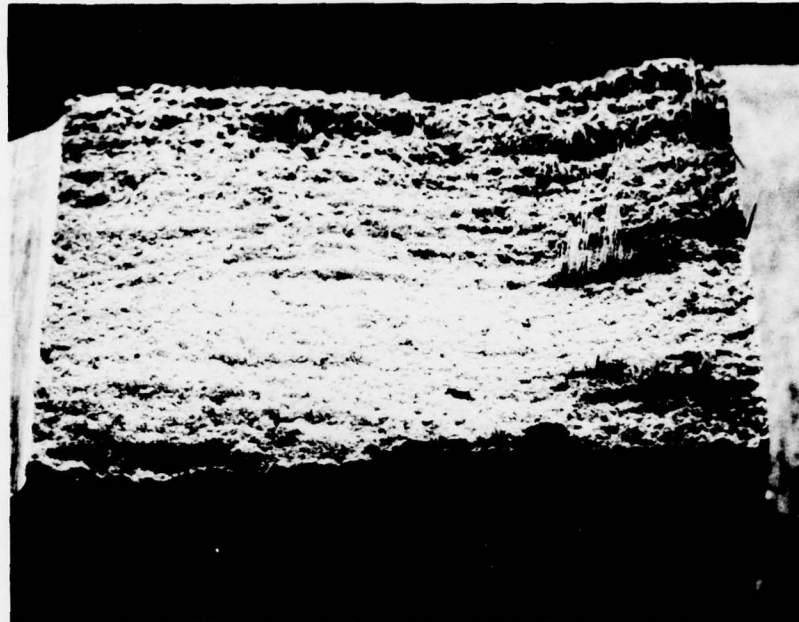
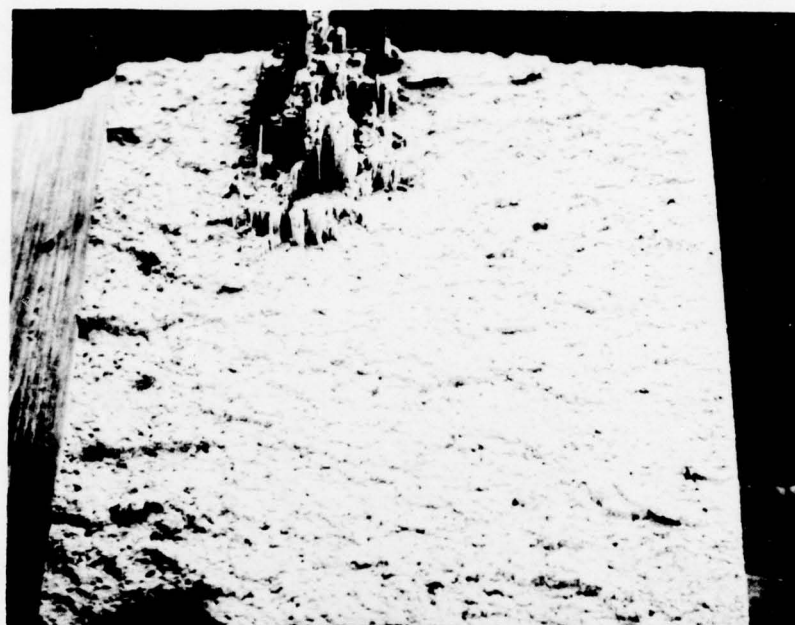
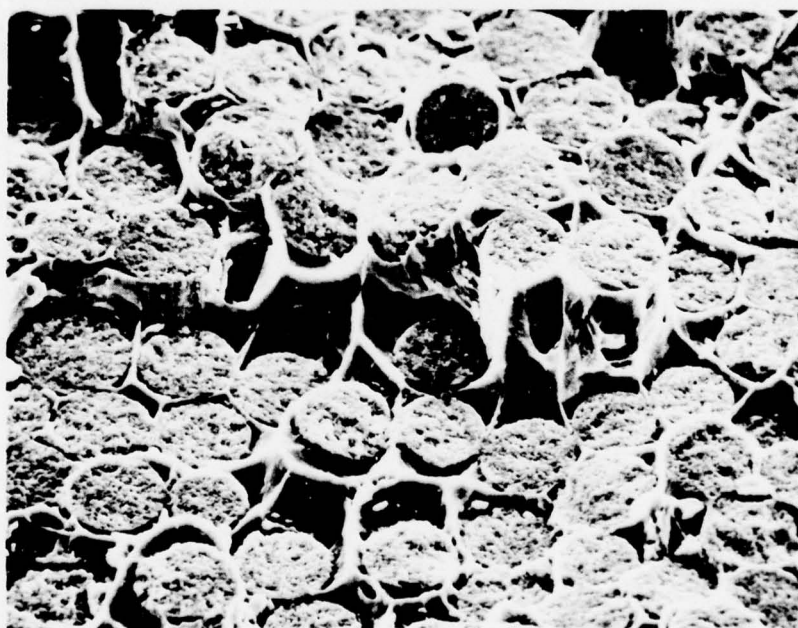


FIG. 19 FRACTURE SURFACE OF CHARPY IMPACT SPECIMEN  
30% SiC YARN REINFORCED Al/Si ALLOY

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1000 $\mu$



20 $\mu$

**FIG. 20 FRACTURE SURFACE OF CHARPY IMPACT SPECIMEN  
60% FP REINFORCED Al/Li ALLOY**



**FIG. 21 FRACTURE SURFACE OF CHARPY IMPACT SPECIMEN  
50% AVCO SiC REINFORCED 6061**



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20. Cont'd

2. The new SiC yarn was characterized as to its surface chemistry, mechanical properties, fracture morphology, and compared with the characteristics of the large diameter monofilament.

3. Powder metallurgical and casting techniques were explored for composite fabrication and it was found that the PM approach offered the greatest success. Low cost, high rate processing techniques were also successfully used to produce high strength composites using both high and low bonding pressures. In the later case the matrix was densified while partially molten.

4. Composite axial strength, transverse strength, and impact energy were measured. The SiC monofilament reinforced composites offered the highest levels of strength and impact resistance; however, the strength of the fiber-matrix bond severely limited composite transverse strength.

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